



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of Masahide Mohri, et al

Serial No.: 09/891,655

Group Art Unit: 1754

Filed: June 27, 2001

Examiner: Steven J. Bos

For: METAL OXIDE POWDER AND METHOD FOR THE  
PRODUCTION OF THE SAME

DECLARATION OF TETSU UMEDA UNDER 37 C.F.R §1.132

Honorable Commissioner of Patents and Trademarks

Washington, D.C. 20231

Sir:

I, Tetsu Umeda, a Japanese citizen residing at 2-13-1, Umezono,  
Tsukuba-shi, Ibaraki, Japan, declare:

That I received a Master Degree from the Graduate School of Kyoto University, Department of Molecular Engineering in March 1990, and entered Sumitomo Chemical Company Limited in April, 1990, in which company I have since then been engaged in research for technology of inorganic material;

That I am familiar with the prosecution history of the above-identified application;

That the following experiment was conducted by me or under my direction.

### **Object of the Experiment**

The object of the present experiment is to show that titanium oxide obtained by a process for making titanium oxide concentrates disclosed in USP 4,517,163 is different from the metal oxide as claimed in the present application.

### **Experimental 1.**

The same method as described in Example 3 in USP 4,517,163 was carried out as follows:

Thirty grams of ilmenite consisting of particles with a size of 50-600  $\mu\text{m}$  (average particle diameter of 280  $\mu\text{m}$ ) and composed of 52.9% of  $\text{TiO}_2$ , 31.7% of iron (Fe) and 0.5% of vanadium (V) was charged into a platinum crucible and then the crucible was placed in an electric furnace with a quartz tube having an inner diameter of 54 mm. The ilmenite particles were calcined under nitrogen gas for one hour at 1000°C. After cooling, the calcined ilmenite particles were heated up to 1000°C while passing chlorine gas through the quartz tube at a flow rate of 30 liter/hr. Gaseous iron(III) chloride evolved. After a reaction period of 3 hours, 16.4 g of yellow flowable powder was obtained. The obtained powder had the peaks of  $\text{TiO}_2$  and the weak peaks of  $\text{Fe}_2\text{TiO}_5$  in a XRD pattern and composed of 85.4% of  $\text{TiO}_2$ , 9.3% of iron (Fe) and 0.7% of vanadium (V). The composition of the powder was determined by X-ray fluorescence spectroscopy. The SEM photographs and the XRD patterns of the raw material ilmenite and the obtained powder are shown in Figs. 1 to 4, respectively.

According to Fig. 3, it was found that titanium oxide thus obtained did not contain polyhedral particles with at least 6 planes each, and a number average particle size of 40  $\mu\text{m}$  or less.

### **Experimental 2.**

Thirty grams of ilmenite consisting of particles with a size of 5-200  $\mu\text{m}$  (average particle diameter of 63  $\mu\text{m}$ ) and composed of 55.8% of  $\text{TiO}_2$ , 30.0% of iron (Fe) and 0.5% of vanadium(V) was charged into a platinum crucible and then the crucible was placed in an electric furnace with a

quartz tube having an inner diameter of 54mm. It was calcined under nitrogen gas for one hour at 1000°C. After cooling, the calcined ilmenite particles were heated up to 950°C while passing chlorine gas through the quartz tube at a flow rate of 50 liter/hr. Gaseous iron(III) chloride evolved. After a reaction period of 2 hours, 15.9 g of yellow flowable powder was obtained. The obtained powder had the peaks of TiO<sub>2</sub> in a XRD pattern and composed of 96.5% of TiO<sub>2</sub>, 0.2% iron (Fe) and 0.8% of vanadium (V). The SEM photographs and the XRD patterns of the raw material ilmenite and the obtained powder are shown in Figs. 5 to 8, respectively.

According to Fig. 7, it was found that titanium oxide thus obtained did not comprise polyhedral particles with at least 6 planes each.

#### Conclusion.

From the results reported above, it is apparent that the titanium oxide obtained by the process disclosed in USP 4,517,163 is different from the metal oxide as claimed in the present application.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above identified application or patent issued thereon.

Date: May 18, 2004

Tetsu Umeda  
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